NSLS-II Planning Workshop:
Earth and Environmental Sciences

Soft x-ray scanning transmission microscopy

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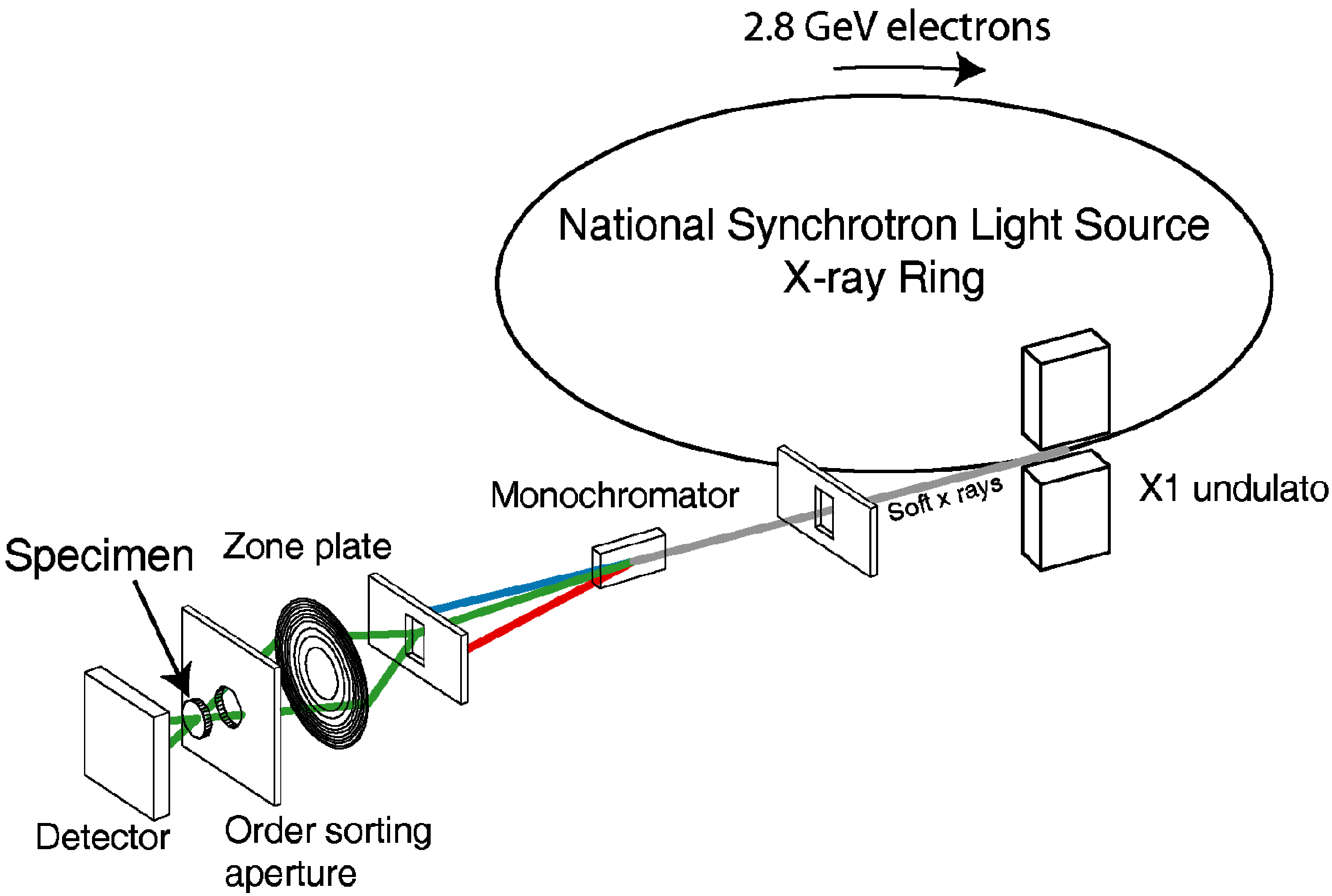
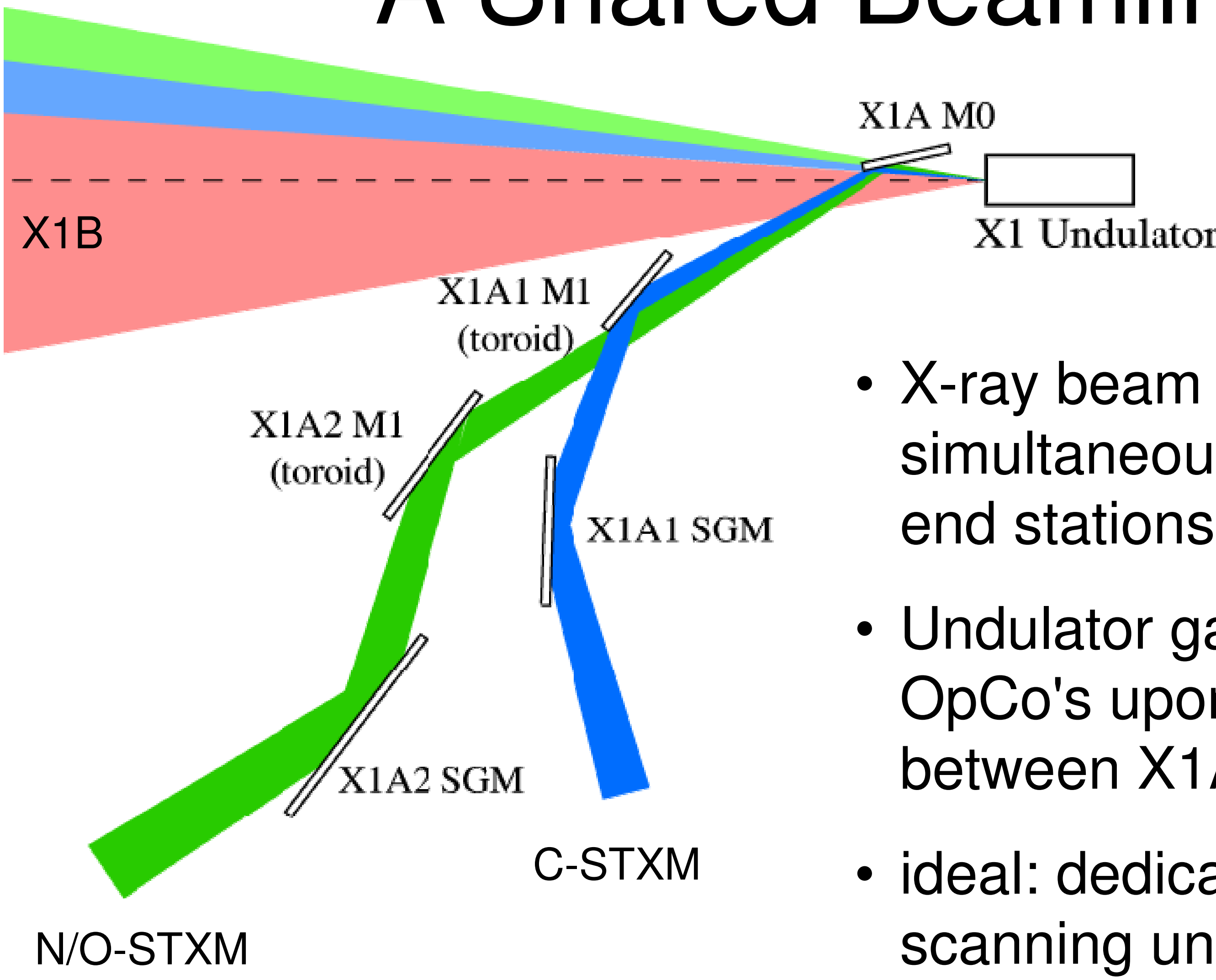
STXM at NSLS X1A



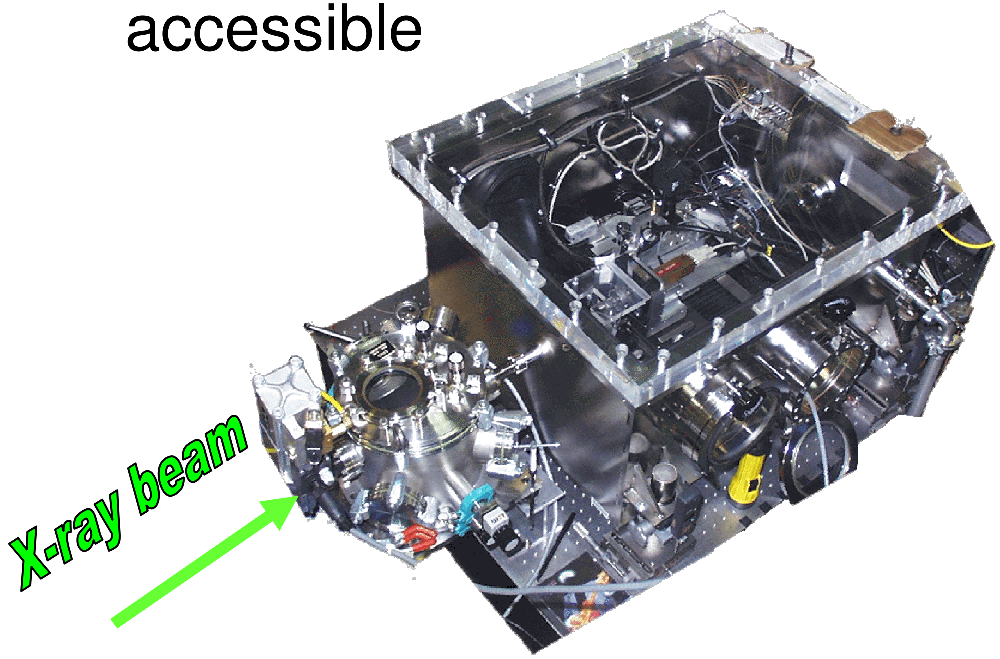
X1
A Shared Beamline

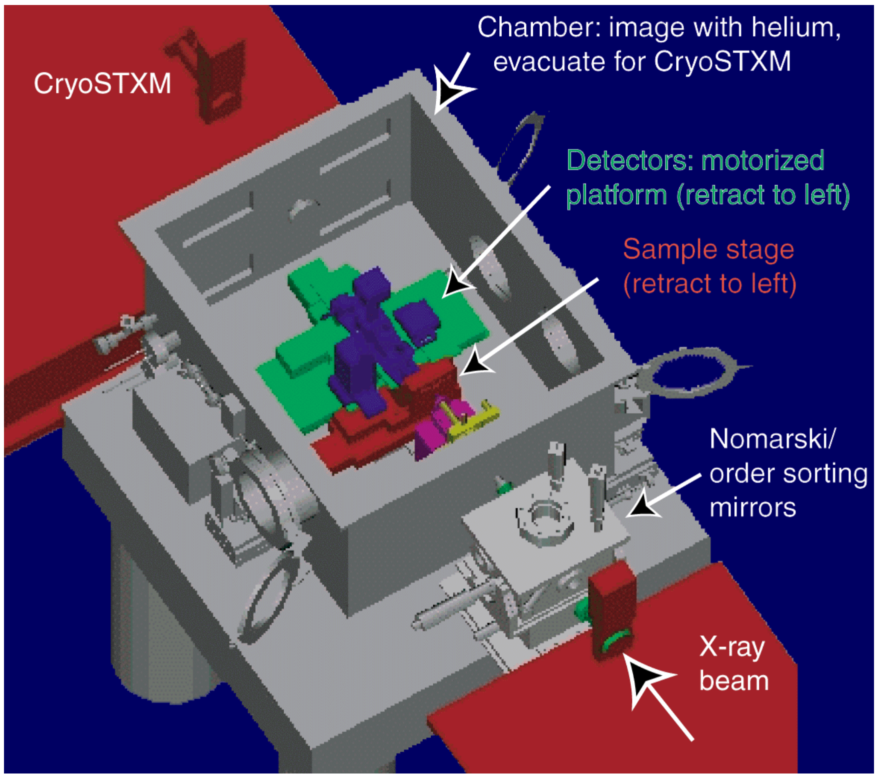


- X-ray beam shared for simultaneous operation of 3 end stations
- Undulator gap controlled by OpCo's upon agreement between X1A and X1B
- ideal: dedicated automatic scanning undulator

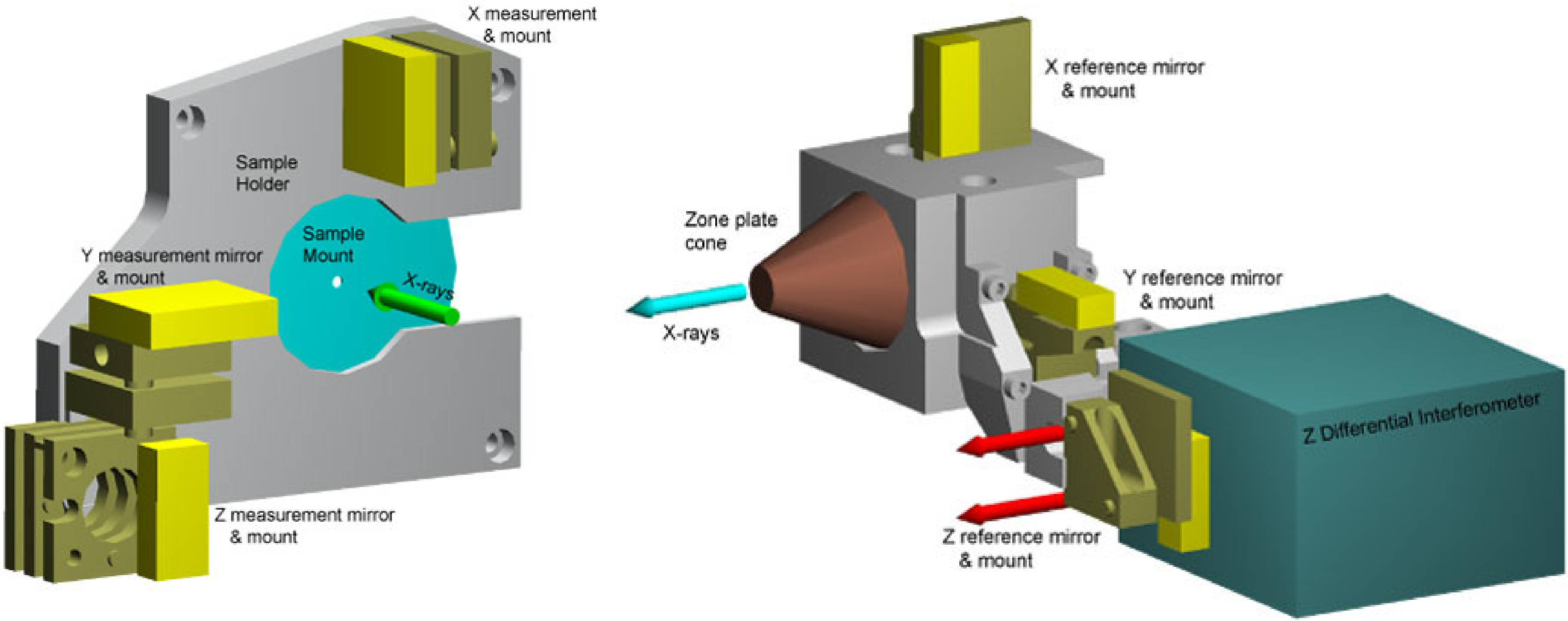
4th Generation Microscope

- Feser, Jacobsen *et al.*, Stony Brook
- Motorized sample and detector platforms
- Sealed, helium-filled chamber: makes E>400 eV accessible





5^{th} Generation Upgrade

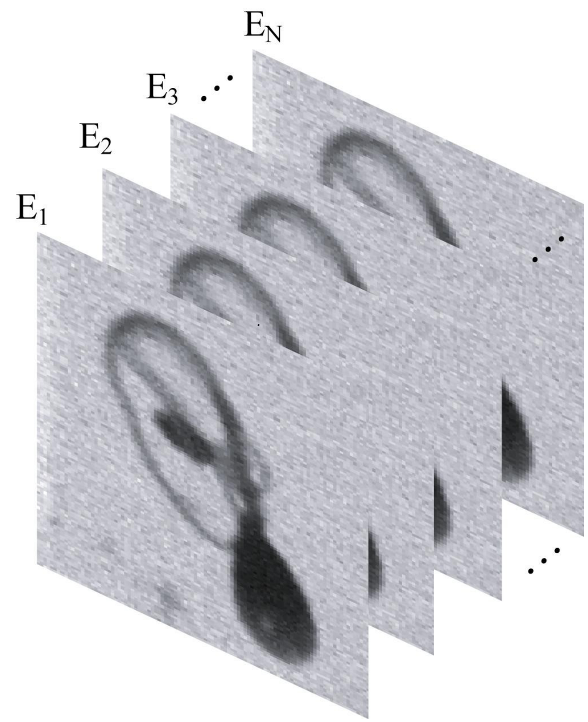


Fast, high resolution scanning necessary

- Differential laser interferometer provides relative position information at 0.3nm resolution
- Automatic position correction in closed feedback loop
- Upgrade of scan and data acquisition electronics

Soft X-ray Spectromicroscopy

- Images with 30nm spatial resolution (~10nm focal spot with conventional zone plates thinkable)
- Series of images over a spectro-scopically interesting energy range (~0.1eV resolution)
- Complex data (~10^5 XANES spectra) ☑ need for sophisticated analysis methods
- Specimen must be stable over ~100 images ☑ cryo preservation

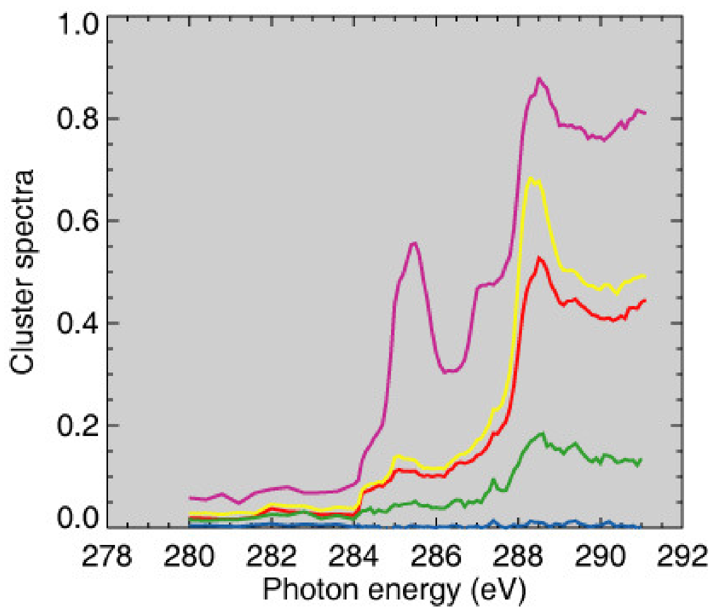


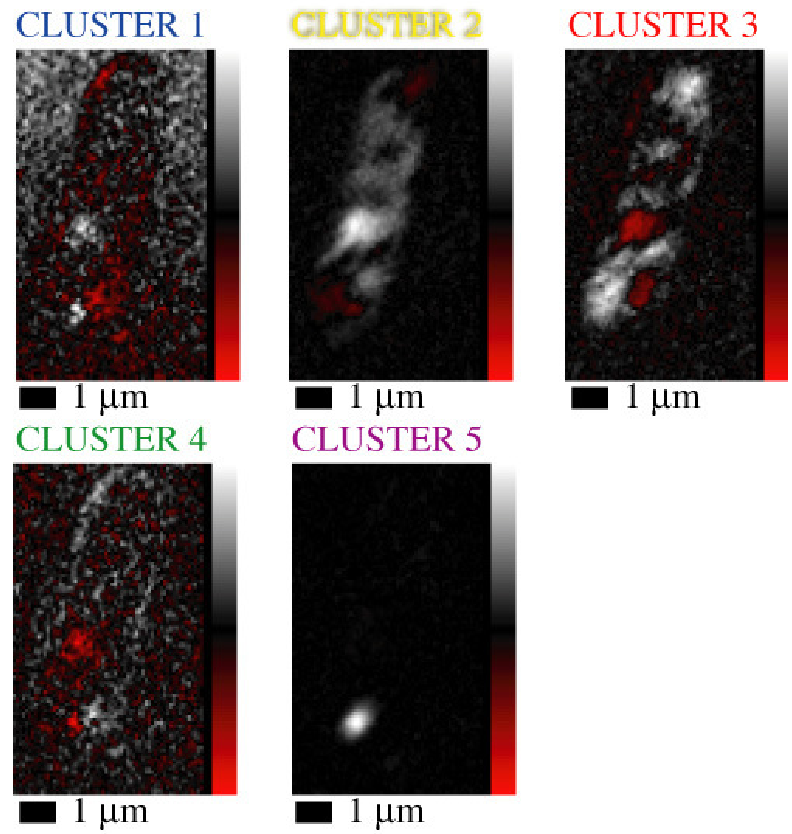
Can be combined with:

- Tomography for 3D chemical speciation!
  Johansson *et al., J. Synch. Rad.* **14**, 395 (2007) at ALS
- Luminescence of quantum dot labels for *pre-selected* proteins (under development)
- …

Cluster Analysis of *Clostridium* Sp.

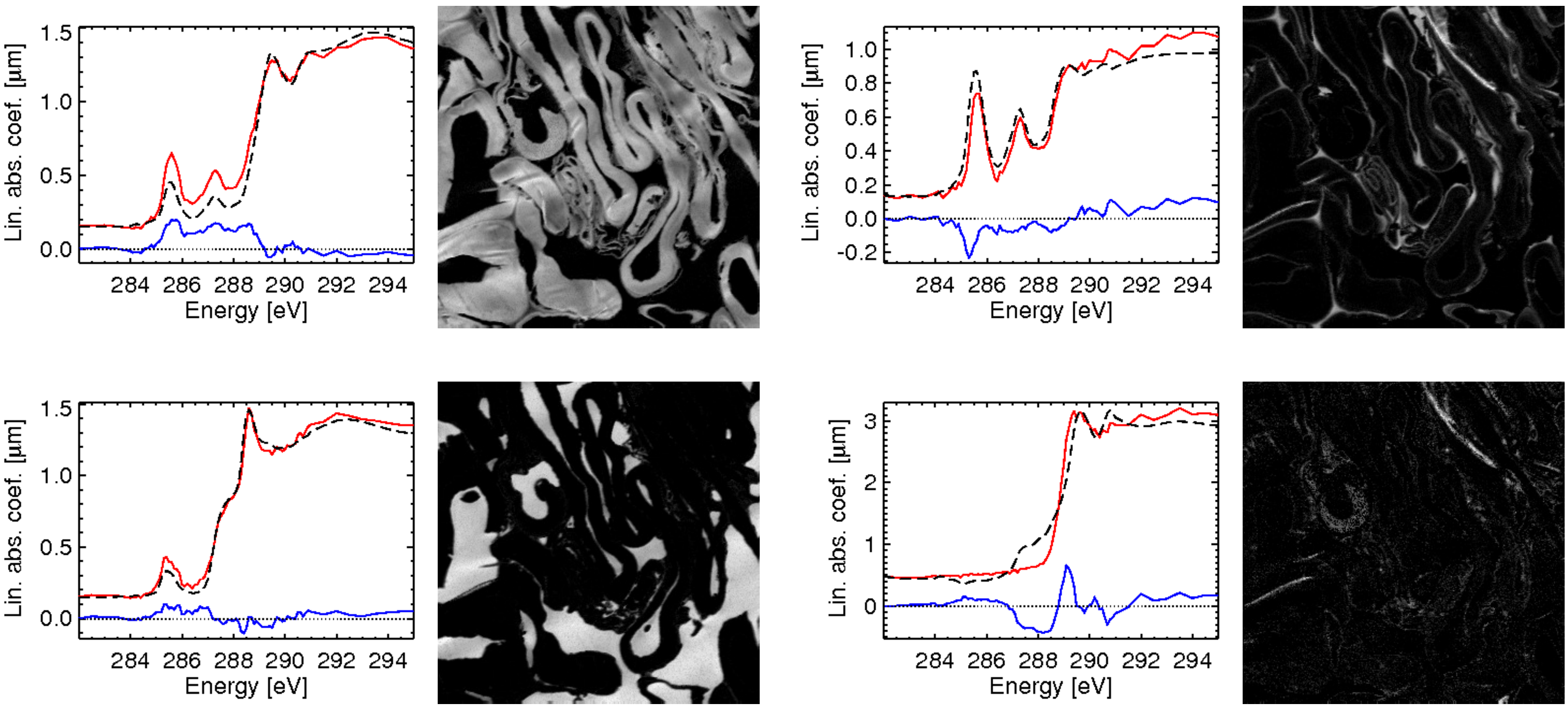
- reveals endospores and intracellular structure (Gillow and Francis)
- cluster analysis, or unsupervised pattern recognition. Lerotic *et al.*, *Ultramicroscopy* **100**, 35 (2005).





Non-negative Matrix Factorization

Wood sample in comparison to reference spectra collected from pure components (wood, LR White Resin, lignin and cellulose)



NMF: Fleckenstein *et al.* (unpublished)
data: Huntsman/Michette

reference, NMF, residual

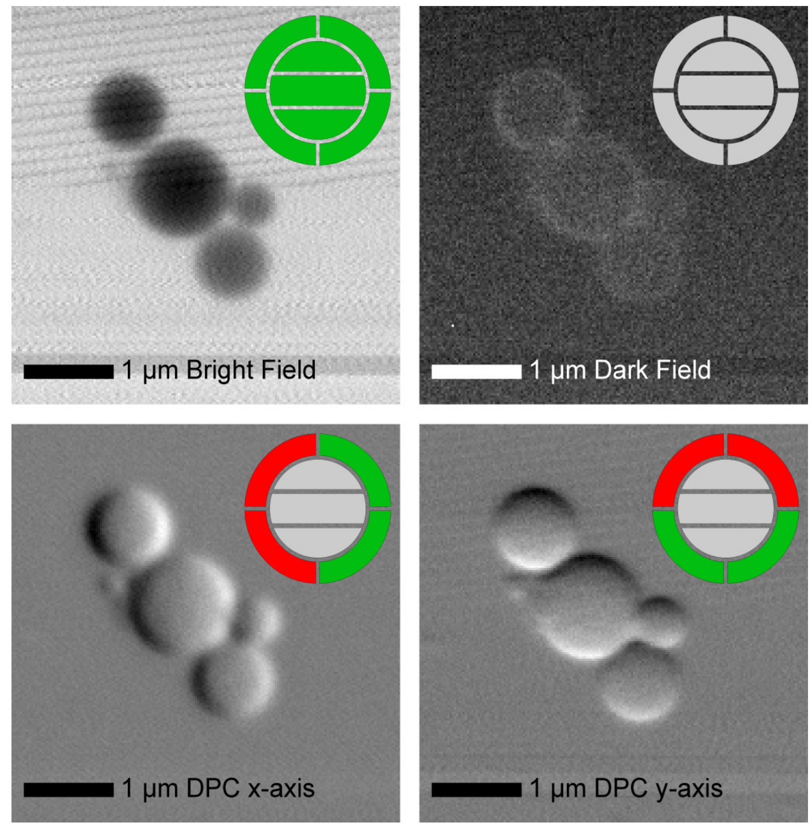
User Demand

- STXM under high demand at NSLS; *very* high demand at ALS; new facilities at SLS; under development at Shanghai, Soleil, Diamond, Australia, BESSY II…
- Present NSLS users mostly from earth and environmental science (Schäfer, Brandes, Christl …) as well as biology
- User suggested improvements:
  - beam stability / low noise
  - faster data collection

    ☑ brightness, motors, detectors, electronics
  - wider energy range (~200-2000eV)
  - high spatial and energy resolution
  - further automation of data acquisition
  - rotatable sample
  - fluorescence capabilities …

Detectors

- Proportional counter
- Phosphor with PMT (ALS STXM)
- Segmented integrating silicon detector
  - For differential phase contrast and dark field imaging
  - High quantum efficiency (>90%)
  - M. Feser *et al.* (Stony Brook)
- Future requirements:
  - high count rates to match higher flux
  - high vacuum compatibility
  - visible light detectors for luminescence studies



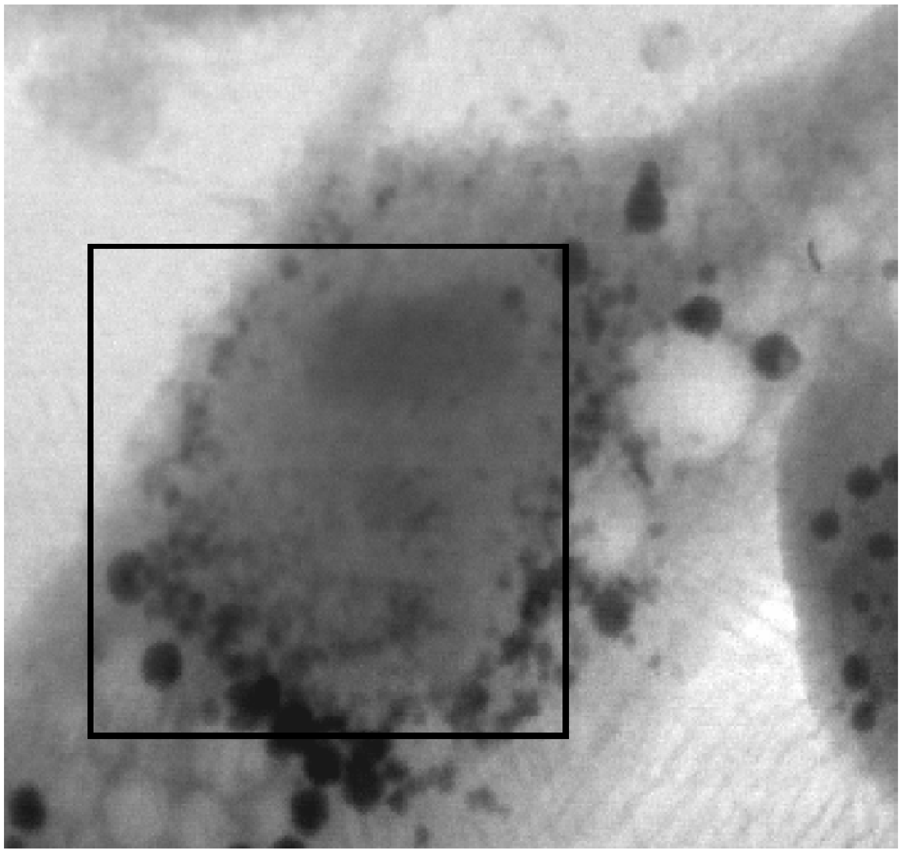
Silica spheres < 1mm

Focusing Optics

- Multilayer Laue lenses
  - hard X-rays only (absorption)
  - difficult to tune (Bragg condition)
- Fresnel lenses
  - limited use for soft X-rays (absorption)
  - chromatic aberration ~ E^2
- Fresnel zone plates
  - first order limit at about 10nm (high aspect ratio)
  - third order possible (with high enough flux)
- High resolution mirrors
  - limit about 10nm below 1keV (Ni or Pt, single-bounce)

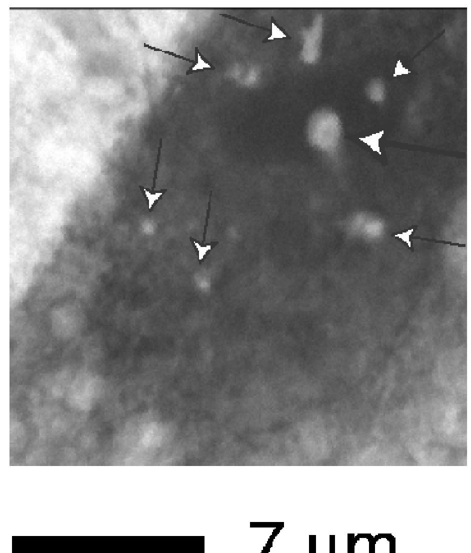
Cryo Preservation

High resolution and flux call for preservation techniques for radiation sensitive samples



Frozen hydrated fibroblast after exposing several regions to ~10^{10} Gray

After warmup in microscope: holes indicate irradiated regions!



7 µm

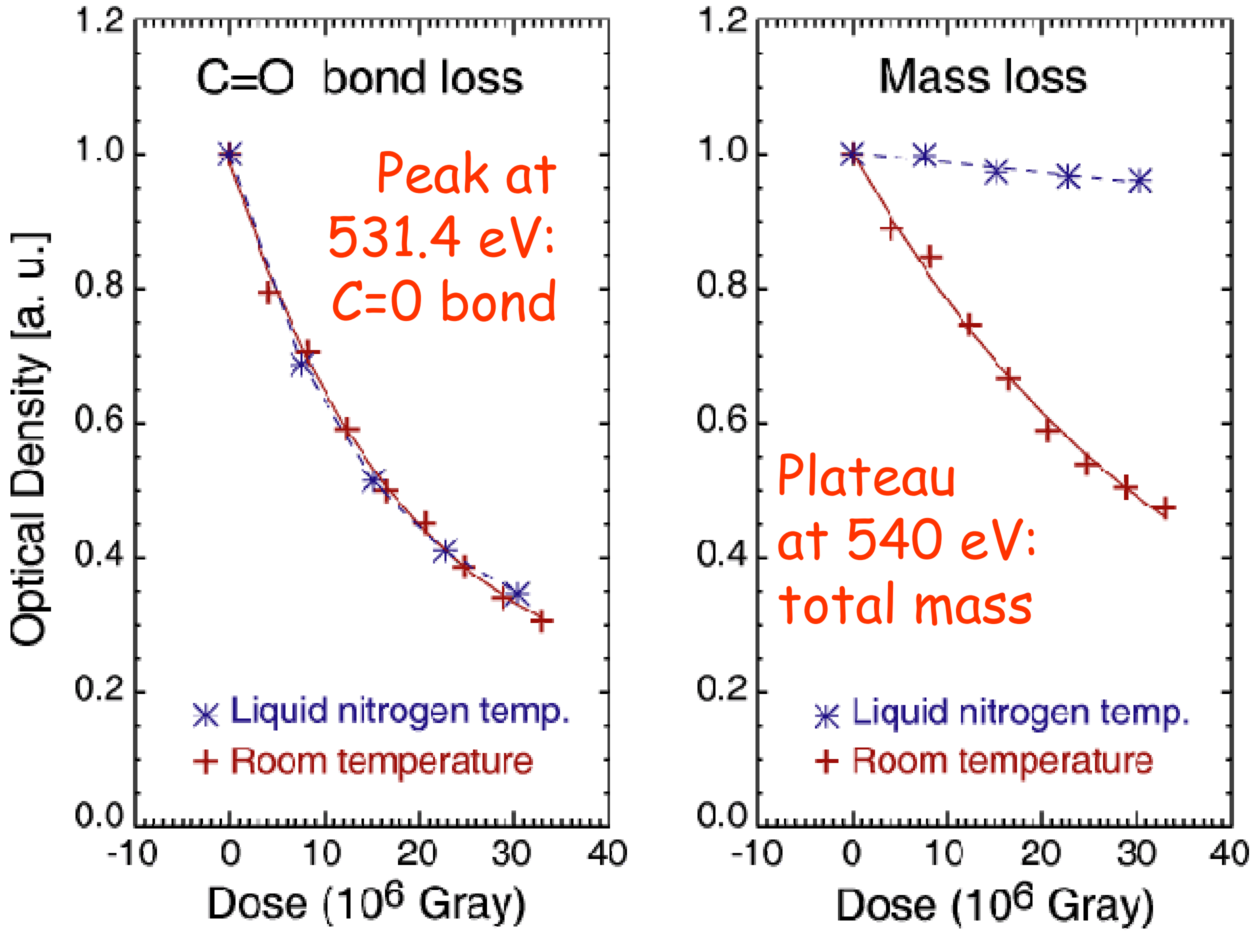
Maser et al., *J. Micros*. **197**, 68 (2000)

STXM at NSLS-II

- Room temperature system at "coherent soft x-ray beamline"
  - atmospheric pressure
  - wet specimens
- System at undulator beamline with cryo capabilities
  - For bio, soft matter, and organic environmental science
  - 100% access
  - Fix location allows for more routine use of complicated system
  - Cryo system for x-ray microscopes under development by Xradia
- Easy transfer of specimens between various instruments (room temperature as well as cryo transfer)
- Sample preparation lab
  - wet cells
  - automatic cryo plunger and high pressure freezer
  - (cryo) ultramicrotome
  - (cryo) light microscope (preselection and quality check)
  - lab x-ray source (check for ice crystallization diffraction rings)

Cryo and XANES

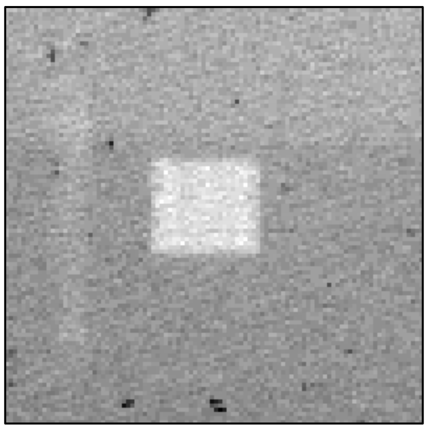
LN_2 temp: protection against mass loss and shrinkage, but not against breaking bonds (at least C=0 bond in dry PMMA)



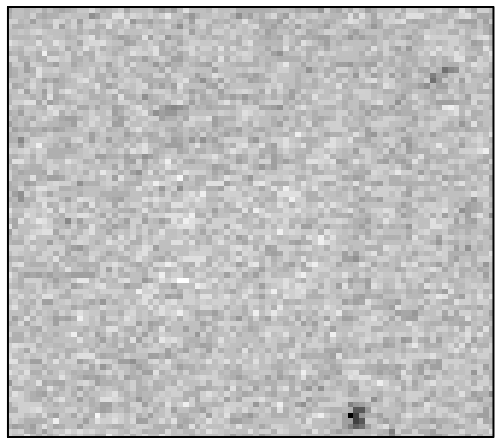
Beetz and Jacobsen, J. Synchrotron Radiation **10**, 280 (2003)

PMMA at room, LN2 temperature

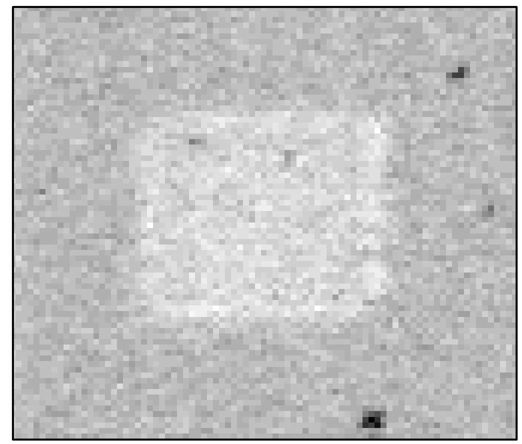
- PMMA: poly methyl methacrylate (plexiglass!) which is especially radiation sensitive – it's used as a resist for electron beam lithography
- Repeated sequence: dose (small square), spectrum (defocused beam). Low dose, larger area image at end.



Room temperature: mass loss immediately visible



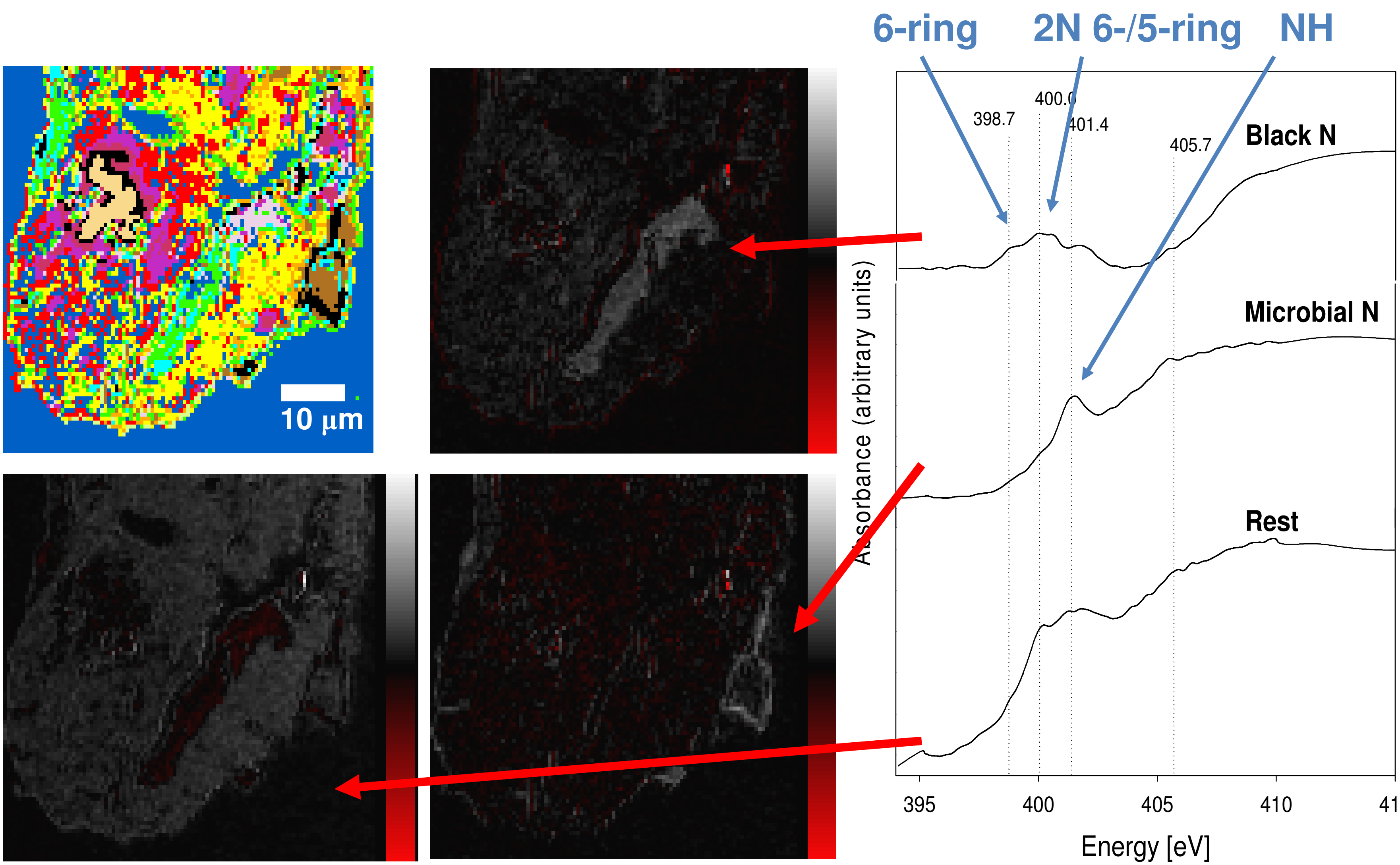
LN2 temperature: no mass loss immediately visible



After warm-up: mass loss becomes visible

Beetz and Jacobsen, *J. Synchrotron Radiation* **10**, 280 (2003)

Spatial Analysis of Nitrogen Forms



Forest Oxisol (Kenya)

J. Lehmann *et al.* (Cornell)